proteins and amino acids, glycerol, and many of the volatile flavor ingredients. Other major additives which are not tobacco identical are essentially similar in composition to natural leaf components. These include materials such as cocoa, licorice, honey, corn syrup, propylene glycol, sugar alcohols, and fruit juice concentrates. By virtue of their compositional and structural similarity to tobacco leaf components, the additives typically used in cigarettes are expected to exhibit similar transfer behavior or to contribute substantially the same types of pyrolysis products to mainstream smoke. Other substances that are used as processing aids such as ethyl alcohol, carbon dioxide, and Freon-11 do not contribute significantly to mainstream smoke composition because virtually none of these materials are incorporated into the tobacco product after manufacture.

BACKGROUND

Cigarette manufacturers and other organizations have conducted and sponsored a significant amount of research into the composition of tobacco leaf and tobacco smoke. This research provides comprehensive information on the contribution of tobacco leaf components to mainstream cigarette smoke. Natural leaf components representing the majority of tobacco leaf dry weight are carbohydrates, proteins, amino acids, organic acids, phenolics, nicotine,

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the tobacco followed by determination of their fates; and (3) study of materials using model pyrolysis test systems.

Each of the above methods can provide valuable information but each method suffers from certain potential drawbacks. For example, use of the first approach (addition of study materials) can yield information which is generally appropriate for additives used at relatively high concentrations (e.g., sugars, humectants, etc.). However, the method is of limited value in quantifying the incremental contribution of an additive to individual smoke constituents where the additive is structurally identical or similar to that of a natural tobacco leaf component. In such cases, it may be difficult to distinguish the additive's contribution to smoke composition from the natural background of smoke components. Also, particular care is needed when amounts of the test ingredient added for investigation are much higher than actual use levels. In such cases, cigarette combustion properties and therefore smoke composition can be altered and the results obtained can be misleading. Additionally, if one adds large amounts of materials in an attempt to study "indigenous" tobacco constituents (e.g., sugars, amino acids, etc.), it is possible that the added materials will not be in the same matrix or state as the indigenous materials; again the pyrolysis results could be misleading.

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same manner. For example, small amounts of triacetin are stripped intact from the filter by mainstream smoke and triacetin also migrates from the filter to the tobacco during product aging. Some amount of this triacetin as well as the triacetin commonly used as a flavor carrier can be volatilized or pyrolyzed during normal smoking. Similarly, the primary components of cigarette paper and adhesives also contribute to mainstream smoke composition, and these materials which include cellulose, lignins, pentosans, citrate and polyvinyl acetate/alcohol were included in the analysis. Table 2 summarizes the list of tobacco components and additives that are included in this study.

Table 3 summarizes cigarette mainstream smoke composition. Cigarette smoke is an aerosol having a discontinuous phase partially comprised of particulate matter and a continuous phase composed in part of vapor constituents. The whole smoke contains nearly 4,000 components.[9,32,43]

APPROACHES FOR INVESTIGATING TOBACCO COMPONENT PYROLYSIS PRODUCTS

Three basic methods are used to study the fate of materials subjected to cigarette pyrolysis. These are: (1) addition of study materials to the tobacco (enrichment/spiking) followed by determination of changes in mainstream smoke composition; (2) addition of radiolabeled study materials to

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Use of radiolabeled additives is an approach that can provide useful data. Radiolabeling experiments, however, are difficult and expensive to conduct and pyrolysis of radiolabeled materials added at higher than actual use levels in diluents / Matrix may still yield misleading results.

Studies using model test systems can give an indication of an additive's potential pyrolysis products under very specific experimental conditions. Such studies are relatively inexpensive to conduct and have the advantage of producing results more rapidly than other approaches. Because of the simplicity in experimental design, however, the studies may not accurately represent the complex thermal, atmospheric, and physical conditions and interactions of actual in-cigarette pyrolysis. For most non-volatile compounds there can be significant discrepancies in the results obtained from laboratory pyrolysis and the types of compounds formed in cigarette smoke. For example, the rate of thermal energy transfer to the substrate in a burning cigarette and the rate at which primary pyrolysis products are removed from the hot zone are conditions that cannot be duplicated under laboratory pyrolysis conditions. Similarly, the presence of oxygen clearly plays a major role in the smoking process, yet pyrolysis in the laboratory is likely to be conducted under oxygenation conditions that differ significantly from that found in the smoking process. In addition, the presence of

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waxes and oils. Many of the major tobacco additives belong to these same class of compounds. The structural similarities between natural leaf constituents and the substances added to tobacco allow conclusions to be drawn on the relationship between cigarette blend components and their contribution to mainstream cigarette smoke.

The vast majority of ingredients added to tobacco -- the flavor ingredients, are highly volatile compounds by virtue of their low boiling points or ease of sublimation. Examples of additives that fall in this category are anethole and trans-anethole, benzyl alcohol, ethyl acetate, and vanillin. This category also includes certain volatile constituents of essential oils such as fenugreek, nutmeg/mace, anise/anise oil and star anise/star anise oil. Because of their physical and chemical properties, these volatile ingredients do not undergo pyrolysis to any significant extent. Rather, they transfer unchanged from tobacco to the smoke at an average rate of 12% to 15% of their applied concentration. Because most additives are applied to tobacco at very low concentrations (<100 ppm) the amounts that transfer to the smoke are minuscule. Other additives which could be added to tobacco, such as the fruit juice extracts, also contain volatile flavor components that exhibit similar transfer and pyrolysis behavior. Moreover, many of the juice extracts, i.e., prune juice and fig juice concentrate, used as

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products are formed from the sugars. The major pyrolyzates are carbon dioxide, carbon monoxide, and water.[21] Small amounts of furfural, 5-hydroxmethyl furfural, 5-methyl furfural, and furan have also been reported.[11, 14, 18, 21, 31, 36, 45] Other reported lesser products include other substituted furans, phenols (especially catechol), volatile aldehydes and ketones, levulinic acid and benzene.[2, 55, 56] It is worth noting that levulinic acid is commonly used as a flavorant in tobacco.

There is good agreement between cigarette enrichment studies, cigarette radiolabel studies and appropriately designed and conducted model system studies on sugar pyrolysis processes. The studies show that the different types of sugars exhibit substantially similar pyrolysis behavior. Glucose, fructose, other hexoses and sucrose yield the same basic pyrolysis products but can differ in some of the relative amounts of individual pyrolyzates formed. There are some minor differences in pyrolysis behavior of the various sugars. Fructose produces more furfural than does glucose or sucrose.[11, 33] Sucrose, reportedly, also produces some maltol.[44a] Pentoses yield pyrolysis products similar to those from hexoses, but with one less carbon for substituted furans.[27]

Polysaccharides. Several polysaccharides are natural components of tobacco leaf. These include cellulose, starch, pectins, dextrins, and pentosans. Several starches,

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cigarette flavoring agents are comprised primarily of sugars and simple carbohydrates (mainly glucose, fructose, xylose and sucrose) and, therefore, their contribution to mainstream smoke composition is expected to be similar to that of the sugars and carbohydrate components naturally found in tobacco.

SELECTION OF COMPONENTS FOR REVIEW

The selection of materials to include in this report is based on a review of the comprehensive literature on tobacco composition. A representative weight percent composition for a typical domestic blend (including additives) is summarized in Table 1.[29] Tobacco leaf is reported to contain over 2500 identified compounds.[9,63]

The major additives used in tobacco products that contribute to mainstream smoke composition were selected for comparison to naturally-occurring tobacco components. Table 1 also summarizes the percent contribution of humectants, casing and flavor ingredients added to a typical domestic blend. Other major additives, such as urea and vanillin, were also included in the set of compounds selected for comparison. Cigarette filter materials could potentially contribute to mainstream smoke if a smoker inadvertently allowed the coal to reach the cellulose acetate filter tow and generate pyrolysis products which might enter mainstream smoke. Additionally, filter plasticizers could be pyrolyzed or volatilized in the

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PRIVILEGED AND [redacted] ATTORNEY WORK PRODUCT

COVINGTON & BURLING DRAFT

April 20, 1993

TOBACCO INGREDIENT PYROLYSIS AND TRANSFER CONTRIBUTIONS TO CIGARETTE MAINSTREAM SMOKE

ABSTRACT

This report summarizes information about tobacco leaf composition and the contribution of tobacco leaf components to the composition of mainstream cigarette smoke, compares cigarette additive composition with tobacco leaf composition, and describes the relationships between cigarette additives and cigarette mainstream smoke composition.

A significant amount of research on the composition of tobacco leaf, tobacco smoke and the contribution of leaf components to cigarette smoke has been conducted over the past 40 years. The research has resulted in the development of a vast amount of information on the contribution of leaf components to cigarette smoke. The information on leaf components can be used to assess the contribution of tobacco additives to mainstream smoke.

Many of the major additives used in cigarettes are identical in composition to naturally occurring tobacco leaf components. Additives which are identical to leaf components are sugars, starches, organic acids and their salts, lipids,

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- Cigarette radiolabel and enrichment studies agree well with appropriate model system studies.

Phenolics. Several phenolics are natural components of tobacco leaf. These include chlorogenic acid (3-(3,4-dihydroxycinnamoyl)quinic acid), lignins, tannins and rutin. A number of the complex natural materials that are added to tobacco (i.e., nutmeg/mace, anise and anise oil, and star anise and star anise oil) are comprised in part of phenolic compounds. As a class the phenolics are characterized by the presence of a phenol component and a non-phenol component (often a sugar or sugar acid). None of these materials is reported to transfer intact to mainstream smoke. The main pyrolysis products reported for this class of materials are noted below:

- Chlorogenic acid yields catechol, catechol derivatives and quinic acid pyrolysis products (sugar breakdown products).[2,18,44,52,69]

- Lignins yield phenols, i.e., catechol plus some guaiacol (methyl catechol) and eugenol (4-allyl-2-methoxyphenol).[18,49,55,56]

- Tannins yield catechols, other phenols, and sugar (usually glucose) breakdown products.[18,44,55]

- Rutin yields products much like those from tannins.[44,52,69]

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Sucrose = Disaccharide

Sugars that are natural components of tobacco include glucose, fructose, sucrose, other hexoses, disaccharides, and pentoses.

These same materials are also used as ingredients in the manufacture of cigarettes. Sugars are major constituents of tobacco casings and are used to adjust the pH of tobacco smoke, thereby reducing irritation and mellowing the taste. Sugars also play an important role in achieving the necessary physical properties of cigarettes. They serve as plasticizers and humectants, thus contributing to the moisture-holding property, elasticity and flexibility of the cut tobacco strands.

Moreover, a number of tobacco additives and flavorings including fruit juices, honey, molasses extract, corn and maple syrups, and caramel are largely comprised of sugars and as such are expected to contribute to smoke composition in the same manner as the sugars naturally found in the leaf. Because the contributions of tobacco leaf sugars to smoke components are well known, this information can be applied to evaluation of the pyrolysis products of potentially new additives composed of similar sugars.

Studies have shown that the major portion of the sugars in tobacco undergo in-cigarette pyrolysis. Only glucose and sucrose are reported to transfer intact to mainstream smoke and even there the transfer rate is very low (roughly 0.5% transfer rate).[11, 12] A wide range of pyrolysis

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transfers into the mainstream smoke, mostly as carbon monoxide, carbon dioxide and ~~other acidic material~~ low organic molecules?.

The main pyrolysis products reported for each of these acids are:

- Some lactic acid transfers intact but the majority decomposes primarily to carbon dioxide and ethanol. In addition some formic acid and acetaldehyde may be formed upon lactic acid pyrolysis.[5,13]
- Oxalic acid may transfer intact through sublimation but mainly it will yield carbon dioxide, carbon monoxide and formic acid upon pyrolysis.[40]
- Malic acid may transfer intact but mainly yields carbon dioxide, carbon monoxide, water, succinic acid, fumaric acid, ethanol and other lesser volatiles.[13,40,67]
- Citric acid pyrolyzes in cigarettes to carbon dioxide, water and citraconic anhydride plus lesser volatiles.[40,48]

Pyrolysis investigations of these materials also indicate that:

- Organic acids will yield primarily carbon dioxide; those with alcoholic hydroxyl groups (i.e., citric, lactic, and malic acids) will also yield water.
- Each organic acid will also yield other predictable pyrolysis products besides carbon dioxide and water.

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Pyrolysis investigations of these materials indicate the following general principles:

- All members of the phenolic class pyrolyze to yield the phenol portion of the material.
- The non phenol portion of each material (often a sugar or sugar acid) yields pyrolysis products expected for their respective chemical classes.
- In-cigarette and appropriate model pyrolysis studies agree well with each other regarding contributions of these materials to mainstream smoke.

Organic Acids. Several organic acids, most notably lactic, oxalic, malic and citric acid are natural constituents of tobacco leaf. Collectively, organic acids may comprise as much as 9 percent of the leaf. A number of these organic acids also are used as additives of tobacco. Lactic acid is added to tobacco in small amounts (up to 0.4%) as a flavorant during the manufacture of cigarettes to give the tobacco a buttery, smooth taste. Citric acid is added to tobacco in the manufacture of cigarettes as a casing, at levels in the range of approximately 1 percent. A number of other organic acid additives, e.g., potassium sorbate, levulinic acid, potassium citrate and tartaric acid, are structurally analogous to the organic acids found in tobacco.

Radioisotope studies on malic, citric and oxalic acids show that only 15 percent of added organic acid

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pectins, and dextrins are also found in certain of the complex carbohydrates that are added to tobacco. Certain of the polysaccharides, such as cellulose and the pentosans, are also major components of cigarette paper. None of these materials is reported to transfer intact to mainstream smoke. Maltodextrin, a simple polysaccharide comprised of D-glucose units that is added to tobacco as a flavorant, also falls within this category. The main pyrolysis products from this group are water, carbon dioxide, carbon monoxide, phenols, organic acids, furan and some substituted furans.[2,14,42,33,53,56,60,61] Other lesser pyrolysis products reported are benzene, toluene, volatile aldehydes and ketones and possibly trace amounts of polyaromatic hydrocarbons.[51,54a,55] This product mix is similar to that from sugars but shows greater emphasis on phenols, aromatics and acids.[31] Pyrolysis investigations of these materials indicate the following general principles:

- All materials studied in this group, except pentosans, yield the same basic pyrolysis products but may differ in some of the relative amounts of individual components.

- Pentosans yield pyrolysis products similar to those from other materials in this group but with one less carbon for substituted furans, for example.

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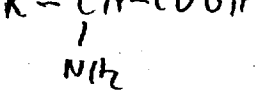
- Cigarette radiolabel studies agree well with appropriate model studies.

Amino Acids and Proteins. Many proteins and amino acids occur naturally in tobacco leaf. A number of amino acids (i.e., L-glutamine and L-asparagine monohydrate) and certain proteinaceous natural materials (i.e., carob bean) are used as additives of tobacco. Amino acids are organic acids containing one or more basic amino groups (NH_2), one or more acidic carboxyl groups (COOH), and another group (R-group) that is the characteristic portion of the particular amino acid. Proteins are comprised of chains of amino acids connected by peptide linkages. A broad range of amino acids and representative proteins was studied in this group. Pyrolysis investigations of these materials indicate that:

- Overall, a protein and a comparable mix of amino acids will yield similar pyrolysis products.[62]
- Particular amino acid R-groups give characteristic pyrolysis products.[1,59]
- Pyrolysis products from cigarette and model studies agree well.

The primary pyrolysis products of proteins and amino acids are reported to be ammonia, carbon dioxide, carbon monoxide and breakdown products of the remaining R-groups (for example phenolics will yield phenols, pyridines will yield pyridines, etc.)[1,14,16,47,57a,62] Other reported products include



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other substances in tobacco are known to modify the pyrolytic pathway, but model test systems do not reflect the interaction of substances that occur in actual in-cigarette pyrolysis.

EVALUATION OF NATURALLY OCCURRING TOBACCO LEAF COMPONENTS

Pyrolysis products of naturally occurring components of tobacco leaf are summarized. The major classes of components are:

- Sugars
- Polysaccharides
- Phenolics
- Organic Acids
- Amino acids and proteins
- Waxes, lipids and oils
- [Nicotine]

Sugars. Several sugars are natural components of tobacco leaf. Sugars can be found in flue-cured and oriental tobacco in amounts ranging up to 33 percent on a dry weight basis, but typically are present in amounts ranging between 5 to 25 percent. Air-cured tobacco such as burley and Maryland types have significantly lower total sugar levels in the maximum range of up to 0.2 percent on a dry weight basis in the cured leaf. The term "sugar" refers to a variety of different, but structurally-related chemical substances.

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TABLE 2

Materials Included in this Review

Sugars
- Glucose
- Fructose
- Sucrose
- Others

Polysaccharides
- Cellulose
- Starch
- Pectins
- Dextrins
- Pentosans

Phenolics
- Chlorogenic acid
- Lignins
- Tannins
- Others

Complex natural materials
- Cocoa
- Licorice

Amino acids and proteins

Waxes, lipids and oils
- Simple esters
- Glyceryl esters
- Acids
- Hydrocarbons

Polyols
- Propylene glycol
- Glycerol
- Sorbitol

Other single materials
- Menthol
- Nicotine
- Triacetin
- Urea
- Propylparaben
- Polyvinylacetate/alcohol

Organic acids
- Lactic
- Maltic
- Oxalic
- Citric

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hydrogen cyanide and other nitriles plus unsaturated aldehydes and acids.[15,25,26] Sulfur containing amino acids and proteins also produce some carbon disulfide.[43a]

In addition to the above compounds, trace amounts of heterocyclic amines have been reported to form as a result of pyrolysis of proteins and amino acids. These include indole types (Trp-P-1, Trp-P-2), quinoxalines (MeIQx), quinolines (IQ), pyridines (PhIP), delta-azacarbolines (Glu-P-1, Glu-P-2) and amino-alpha-carbolines (A alpha C, MeA alpha C).[30,35,36,37,68]

Waxes, Lipids and Oils. Many waxes, lipids and oils occur naturally in tobacco leaf.[6] These include simple long chain esters, glyceryl esters, long chain fatty acids and long chain hydrocarbons. Methoprene, a pesticide used during processing to protect stored tobacco from the cigarette beetle, is a member of this class of compounds. Pyrolysis investigations of these materials indicate that:

- Esters and acids will generally yield carbon dioxide during pyrolysis.[38]
- The hydrocarbon chains reportedly undergo scissions producing either lower molecular weight alkanes, alkenes and some aromatics if behind the coal with little or no oxygen or volatile aldehydes and ketones if appreciable oxygen is present.[6,17,38]
- Some of these materials transfer intact into the mainstream smoke particulate phase.[67]

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TABLE 3

Mainstream Smoke Composition for a Representative U.S. Blend Full Taste Filtered Cigarette

flavor?

Whole Smoke	% By Weight
N_2	62 X
O_2	13
CO_2	12.5
CO	4
H_2O*	2
H_2	1
Particulate phase**	4
Vapor phase***	1.5
	100.0

* From particulate + vapor phase
** No H_2O; 17.6 mg tar + 1.4 mg nicotine
*** No H_2O; less the above gases; ~6.8 mg.

Particulate and Vapor Phase Compositions

Constituent or Class	mg In Particulate Phase	mg In Vapor Phase	mg Total	Number of Identified Compounds
Hydrocarbons	3.15	3.04	6.19	760
Aldehydes	1.58	1.35	2.93	110
Ketones	0.90	0.81	1.71	520
Nitrogen heterocycles	0.79	0.14	0.93	920
Esters	0.79	0.07	0.86	480
Organic acids	2.93	0.10	3.03	230
Alcohols	1.80	0.14	1.94	380
Nicotine	1.35	---	1.35	1
Phenols	0.79	---	0.79	280
Nitriles	---	0.61	0.61	110
Others	4.84	0.49	5.33	Hundreds
Totals	18.9	6.8	25.7	~3800

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Materials sharing common chemical features yield similar pyrolysis products but relative amounts of individual components may differ. Thus, pyrolysis studies of cigarette additives and tobacco leaf components of similar chemical structure and/or composition yield similar pyrolysis products.

The approach of using compositional analogy for tobacco leaf components and additives provides a sound technical basis for predicting the potential pyrolysis products of proposed new tobacco additives.

Addition of volatile aromatic flavor substances is expected to impart specific flavor notes by virtue of their intact transfer into tobacco smoke. The concentrations of these ingredients, however, is extremely small and they generally are of similar chemical classes that occur naturally in tobacco and tobacco smoke.

While inclusion of non-volatile additives is/are expected to ~~make~~ cause small changes in the relative amounts of individual smoke components or classes of smoke components, these changes are not expected to significantly depart from the amounts or types of components generated from a range of additive free tobaccos or tobacco blends.

Attachment

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TABLE 1

% Chemical Composition of a Representative U.S. Cigarette Blend (Tobacco Plus Additive)

	Total % By Weight	% From Tobacco	% From Additives
Carbohydrates			
Sugars	12.0	8.0	4.0
Cellulose	9.5	9.5	<0.1
Pectins	8.5	8.5	<0.1
Starch, pentosans	2.5	2.5	<0.1
Proteins	6.0	6.0	<0.1
Amino acids	2.5	2.5	<0.1
Organic acids	9.0	9.0	<0.1
Phenols	6.5	6.5	<0.1
Lignins	3.5	3.5	<0.1
Nicotine	2.0	2.0	----
Volatile bases	2.0	2.0	----
Waxes, resins	8.0	8.0	<0.1
Humectants & other additives	6.0	---	6.0*
Water	12.0	12.0	----
Inorganics	6.5	6.5	----
Others	3.5	3.5	----
Totals	100.0%	90.0%	10.0%

* Humectants & other additives

Humectants	3.5
Cocoa	0.9
Licorice	0.9
Menthol	0.2
"Ammonia"	0.5
Volatile flavorants	<0.1
Subtotal	6.0%

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Complex Natural Materials. Cocoa and licorice were reviewed in this group. The following results have been reported in the literature:

- Cocoa added to tobacco at levels of 2-4 percent reportedly results in a slight increase in tar, phenols, ammonia and selected other mainstream constituents.[65] Typically, cocoa used in cigarettes is about one-fourth butterfat, and as noted earlier, some of these materials can transfer intact into mainstream smoke particulate phase.[67] The composition of cocoa is well documented.[16a] Cocoa powder contains more than 300 volatile compounds, many of which are produced during the roasting process. Many of those compounds also occur in smoke from cigarettes to which cocoa has not been added.[16b]

Cocoa powder was pyrolyzed in a nitrogen atmosphere at temperatures from 370 to 750°C and the pyrolyzates were analyzed.[54b] Cocoa powder produced palmitic and stearic acids on pyrolysis, particularly at 350 and 450°C, probably arising from thermal degradation of cocoa butter triglycerides. The author concluded that phenols derived from pyrolysis of cocoa powder should not significantly enhance the phenol content of tobacco smoke, although the fatty acid composition of smoke might be affected by triglyceride breakdown products.

The addition of cocoa to cigarettes, over a wide range of concentrations, has a minimal affect on the chemical

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Sear

pyrolysis products reportedly include furans (from sugars), phenols (e.g., guaiacol) and glycerol plus a small amount of glycyrrhizic acid.[30,66] The fate of glycyrrhizic acid and glycyrrhetinic acid during the smoking of cigarettes has been studied by adding these acids separately to cigarettes.[30] It was found that glycyrrhizic acid decomposed to glycyrrhetinic acid and was transferred in the mainstream smoke as such. ? M_W 470 ! When glycyrrhetinic acid itself was added to cigarettes, it mp 296 was transferred intact to the mainstream smoke. In both cases, the amount of glycyrrhetinic acid found in the smoke condensate was small, and it was concluded that the glycyrrhizic acid in licorice root used for tobacco flavoring was mostly decomposed on smoking.

Several low level volatile materials occurring naturally in cocoa and licorice have been reported to transfer intact to smoke.[16a,30,66]

Processing Aids. Ethyl alcohol, carbon dioxide and Freon-11 are used as processing aids in the manufacture of cigarettes. Because of their extremely high volatility and the manner in which they are used in the manufacturing process, virtually 100 percent of these materials are lost through volatilization and they are not incorporated into the final tobacco products.

In the case of Freon-11, a substance used as a processing aid to expand tobacco, the high temperatures used

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14-20%, depending on cigarette design. The distribution of ^{14}C-menthol (uniformly labeled) and its combustion and pyrolytic products in cigarette smoke have been studied. The mainstream smoke contained 28.9% of the total activity, with 44.3% in the sidestream smoke, and 26.9% in the butts. The major ^{14}C-menthol smoke product in the mainstream smoke was unchanged menthol (98.9%). Minor components that were identified in the mainstream smoke were carbon dioxide (0.1% of total mainstream activity), menthane (0.2%), and menthone (0.4% present as a contaminant of the labeled menthol). The additional 0.4% of the mainstream activity was located in unidentified peaks in the gas radiochromatograph.

Cigarettes were labeled with ^{14}C-menthol, smoked, and the fate of the menthol was followed using an inverse isotope dilution assay. The mainstream smoke contained 41.87% of the radiolabel with 41.41% being in the solids and 0.46% in the gases (CO, CO_2, other gases). Unchanged menthol accounted for approximately 96% of the label in the mainstream solids. The sidestream smoke contained 37.56% of the label with 33.62% being in the solids and 3.94% in the gases (CO, CO_2, other gases). Unchanged menthol accounted for approximately 92% of the label in the sidestream solids. An additional 20.49% of the label was recovered in the butts and filters and 0.07% in the ashes. The author only investigated the amount of unchanged menthol present in the mainstream and sidestream

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than 1% of the cigarette tobacco by weight. Urea reportedly lowers cigarette gas phase delivery across the board but does not alter relative deliveries of HCN, nitrogen oxides, carbon monoxide or aldehydes.[46] Model studies report urea pyrolysis products to be water, ammonia and carbon dioxide.[33] Urea doesn't appear to transfer intact into cigarette smoke.

One study has reported that urea when added to tobacco reduces the amount of certain pyrolysis products in smoke: 43% decrease in benzo[a]pyrene; 15% decrease in CO; and 20% decrease in crude condensate.[43b] In addition, the study indicated that urea impedes the formation of N-nitrosamines.

- Propylparaben (propyl-parahydroxy benzoate) is used in the manufacture of reconstituted tobacco to prevent spoilage. Chromatographic (GC/MS) analysis of propylparaben pyrolysis indicates that at 315°C, the material distills without pyrolytic cleavage. At 590°C a very small amount of phenol is generated, but the vast majority of the chemical transfers unchanged.

z. B. Lebrauch als Bindemittel in Farben

?

- Polyvinyl acetate is reported to yield acetic acid upon pyrolysis.[19a] Polyvinyl acetate and polyvinyl alcohol are reported to yield common products including acetone, acetaldehyde, water and some crotonaldehyde.[3,4,8]

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Nicotine. Nicotine is reported to transfer intact into mainstream smoke at about 8-10% of blend concentration for a full flavor filtered cigarette.[20] Pyrolysis products of nicotine are reported to include mainly carbon dioxide and several pyridines, ~~primarily pyridine~~ (3-methyl pyridine ~~and~~, 3-vinyl pyridine etc.)[20,58]

Appropriate nicotine pyrolysis model studies agree well with in-cigarette pyrolysis results.[28,58]

EVALUATION OF CIGARETTE ADDITIVES

Polyvalent alcohols ~~Polyols~~. ~~Polyols refer to~~ = diols Polyvalent ~~polyhydric~~ alcohols are compounds containing two or more hydroxyl groups. Those having three hydroxyl groups (trihydric) are glycerols, those with more than three are called sugar alcohols. Several ~~polyols~~ are added to tobacco. These include propylene glycol, glycerol, and the hexahydric sugar alcohol, sorbitol. Maltitol, a polyhydric alcohol used as a humectant and a sweetener, also is a member of this class of materials.

- Propylene glycol has been used in cigarettes for thirty-five years as a humectant and as a vehicle for the addition of flavors. A typical cigarette may contain 1.5 to 15 mg of propylene glycol, or about 0.2 to 2 percent of the cigarette tobacco by weight. Pyrolysis studies have shown that most ~~only a small portion~~ of the propylene glycol added to cigarettes, (from 5 to 12.6 percent) is transferred intact to

is retained in the butt and

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in the expansion process causes the Freon-11 to volatize. Approximately 99 percent of the Freon-11 is recovered in the process and residual levels in the cigarette are less than 5 micrograms/cigarette. Ethyl alcohol, on the other hand, occurs naturally in tobacco smoke, at a level of approximately 2 micrograms/cigarette. Although it also is used in cigarette manufacture as a solvent or carrier for flavor ingredients, essentially all of the added ethyl alcohol is volatilized during the manufacturing process, and the smoke contains virtually no incremental level of ethyl alcohol.

Carbon dioxide is formed naturally during cigarette smoking, and accounts for roughly 13 percent of all mainstream smoke. The carbon dioxide used in the manufacture of cigarettes is lost through the volatization processes and virtually none of it remains in the tobacco.

CONCLUSIONS

There are three primary approaches for investigating the contributions of tobacco leaf components and cigarette blend additives to cigarette mainstream smoke. Each has its unique strengths and limitations.

Despite these limitations, results from laboratory simulation studies generally agree with in-cigarette test results when test conditions are comparable for temperature, heating rates, and ambient gas compositions.

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Particle phase condensate

~~solids~~ and did not ~~present data on~~ any pyrolysis products of menthol ~~in these solids~~.

These reports may be contrasted with model test studies of the pyrolysis of pure menthol.[37b] Menthol was pyrolyzed in horizontal quartz tubes packed with quartz chips under a stream of dry nitrogen at 600 and 860°C. Significant amounts of menthol were found to be pyrolyzed under these conditions -- 22% at 600° and 84% at 860°. Pyrolysis products included benzene, toluene, and styrene. In contrast, the studies described above which tracked the fate of ^{14}C-labeled menthol added to tobacco found that well over 90% of the menthol remained in the smoke or the butt as unchanged, unpyrolyzed menthol. This suggests that pyrolytic studies [of pure compounds] may be poor predictors of what actually occurs when the compound is burned in the cigarette. This is particularly true when test conditions are not comparable for temperature, heating rates, and ambient gas compositions within a burning cigarette.

- Triacetin transfers almost completely intact from the filter [and tobacco section]. A small portion of triacetin, following diffusion to ~~on~~ the tobacco section, has been reported to pyrolyze to acetic acid, glycerol, acetone, acetin and diacetin.[46]

- Urea is a natural component of tobacco, and it is also added to tobacco as a flavorant. A cigarette to which urea has been added may contain 2.2 to 5.4 mg of urea, or less

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composition of smoke. Tests on cigarettes cased with a variety of materials showed that, as the cocoa content of the casing mixture increased from 1.5 to 7.5 g/100 g of tobacco, the tar content per cigarette increased from 19.2 mg to 19.7 mg; the benzo[a]pyrene from 10.3 nanograms to 11.6 nanograms; phenol from 45 micrograms to 48 micrograms; ammonia from 36 micrograms to 49.5 micrograms; and benzo[a]pyrene/TPM from 0.43 nanograms/mg to 0.48 nanograms/mg. Other components of smoke that were measured either decreased, or remained the same, ~~or were variable~~.

In another study, cocoa bean hulls were formed into thin sheets that were made into cigarettes. Levels of pyrene, fluoranthene, benzo[b]fluoranthene, and benzo[a]pyrene in smoke condensate from these cocoa "cigarettes" were not as high as the maximum levels of these compounds in tobacco cigarettes.[19b]

- Licorice is used in cigarettes both as a flavor and as a casing material to smooth the harsh taste of certain kinds of tobacco. Most cigarette tobacco blends contain less than 1 percent licorice. Licorice contains from 6 to 14 percent of the ~~tribasic acid~~ glycyrrhizic acid, also known as glycyrrhizin. Besides glycyrrhizin, licorice contains sugars, starch, plant gums, resins, flavonoids, essential oils, inorganic salts and low levels of nitrogenous constituents such as proteins, amino acids, and nucleic acids.[10,39] Licorice

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